भारतीय मानक
Indian Standard

IS 8628 : 2020

जी साल्ट, तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

G Salt, Technical — Specification

(Second Revision)

ICS 71.080.40

© BIS 2020

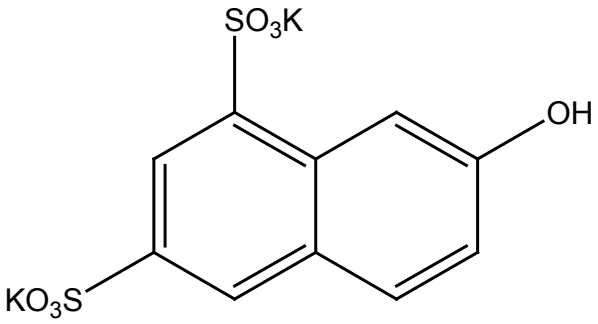
भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

August 2020 **Price Group 4**

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

G Salt (G10H6O7S2K2), chemically described as 2-naphthol-6, 8-disulphonic acid is a dye intermediate. It is available as its dipotassium salt and is represented by the following structural formula:



G Salt
(Molecular Mass, Free Acid 304)

This standard was first published in 1977 and revised in 1987. In the first revision, the requirement of assay (on the basis of molecular mass 304), β-naphthol and matter insoluble in sodium hydroxide were modified. Besides, the requirement of impurities, namely, R salt and Schaeffer's Salt were stipulated and chromatrographic test method for their estimation were introduced.

Considering to the development in analytical techniques and use of more sophisticated instrument to determine purity and impurity profile, the committee has decided to revise the standard. In this revision, test method for Purity and impurities like β-naphthol, R-saltand Schaeffer's Salt content using High Performance Liquid Chromatography (HPLC) have been incorporated.

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

G SALT, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for G salt, technical**.**

2 REFERENCES

The following Indian Standards contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
2552 : 1989	Steel drums (galvanized And Ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off-white powder or off-white moist cake.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Tare, net and gross mass.

4.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.'

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Tests for assay shall be conducted on each of the individual samples.

5.2.2 Test for the determination of remaining characteristics, namely, β-naphthol; R Salt and Schaeffer's Salt content and solubility in sodium hydroxide solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **5.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE –'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirement for G-Salt, Technical

(*Clause* 3.2)

Sl No	Characteristic	Requirement	Method of Test
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), *Min*	65	**A-2**
ii)	Matter insoluble in sodium hydroxide solution, percent by mass, *Max*	0.2	**A-3**
iii)	Purity, percent, by HPLC, *Min*	65	B
iv)	β-naphthol content (on 100 percent basis), percent by mass, *Max*	0.5	C
v)	R Salt, percent by mass (on 100 percent basis), *Max*	1.0	C
vi)	Schaeffer's salt, percent by mass (on 100 percent basis), *Max*	1.0	C

ANNEX A

(*Table* 1 *and Clause* 6. 1)

METHODS OF TEST FOR G SALT, TECHNICAL

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 105°C and 1°C to constant mass. Grind and mix well. Transfer the material immediately to a wide-mouthed bottle and stopper it. Use this prepared sample for tests.

A-2 ASSAY

A-2.0 Outline of the Method

The material is distilled in dilute sodium carbonate solution. A known volume of the solution is titrated against standard 4-nitrobenzene diazo solution in alkaline medium and from the consumption diazonium solution, assay is calculated.

A-2.1 Reagents

A-2.1.1 *Sodium Carbonate Solution***,** *approximately 10 percent (m/v).*

A-2.1.2 *Phenolphthalein Indicator Paper*

A-2.1.3 *Standard 4 Nitrobenzene Diazo Solution ─ 0.1N.*

A-2.1.4 *H Acid Indicator Solution*–Dissolved 0.5 g of H acid in 100 ml of 1 percent ammonium hydroxide solution

A-2.2 PROCEDURE

Weigh accurately about 15 g of the prepared sample (*see* **A-1.1)** and transfer to a 500 ml beaker with the help of water. Slowly add sodium carbonate solution to get a positive test on phenolphthalein in paper. Transfer the solution quantitatively into a 500 ml volumetric flask and dilute to the mark with water at room temperature. Mix well. Pipette 50 ml aliquot of this solution into a 1 litre beaker. Add 200 ml of ice-cold water. Add 50 ml of sodium carbonate solution. Stir with a mechanical stirrer and cool with washed ice to below 1°C.Titrate with 4-nitrobenzene diazonium solution from a cold water jacketed burette. Test with H acid for excess of diazo and with the diazo for the coupling component. The end point is noted when there is no test with diazo and a fine visible pink line with H acid indicator is obtained which may persist for 10 min without further addition of diazo. Let the titre reading be *V*.

A-2.3 CALCULATION

A-2.3.1 Assay, percent by mass **(**on dry basis) =

$$\frac{V \times N \times 304}{M}$$

where

V = volume of the standard 4-nitrobenzene diazonium solution, in ml;

N = normality of 4-nitrobenzene diazo solution; and

M = mass (on dry basis) of the material taken for the test, in g.

A-3 DETERMINATION OF MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

A-3.1 Reagent

A-3.1.1 *Sodium Hydroxide Solution* ─ Approximately 5 percent (*m/v),* filtered free from suspended impurities.

A-3.2 Procedure

Weigh accurately 10 to 15 g thoroughly mixed sample into a 1 000 ml beaker, added 300 ml water and sufficient 5 percent sodium hydroxide solution to make the solution alkaline to brilliant yellow paper. Heat the solution to 60°C until the sample is dissolved and filters it through sintered crucible of porosity G_4, wash residue well with hot water, dry at 100 and 5°C, cool and weigh.

A-3.3 Calculation

Matter insoluble in sodium hydroxide solution, percent by mass = $\frac{M_1 \times 100}{M_2}$

Where

M_1 = mass of the residue, in g; and

M_2 = mass of the sample taken for the test, in g.

ANNEX B

[*Table* 1, *Sl No. (iii)*]

TO DETERMINE PURITY OF G SALT BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-0 OUTLINE OF METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-1 OBJECTIVE

To determine Purity of G Salt by high performance liquid Chromatography

B-2 APPARATUS

Binary Gradient Liquid chromatography system with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-3 COLUMN

C 18 100A 250 × 4.6 mm, 5 µm or equivalent

B-4 REAGENT :

1 Acetonitrile – HPLC grade
2 Water – HPLC Grade
3 Tetra Butyl ammonium Hydrogen sulphate-HPLC Grade
4 G Salt-Known Purity Standard

B-5 STANDARD PREPARATION

Weigh accurately 0.050 0 gm standard G Salt in 100 ml volumetric flask dissolve it in Acetonitrile & make up to the mark with Acetonitrile.

B-6 SAMPLE PREPARATION

Weigh accurately 0.050 0 gm Sample in 100 ml volumetric flask dissolve it in Acetonitrile & make up to the mark with Acetonitrile.

B-7 BUFFER PREPARATION

0.2% Tetra butyl ammonium hydrogen sulphate in HPLC grade Water.

B-8 FLOW RATE

1.00 ml/min

B-9 MOBILE PHASE

Acetionitrile	Buffer
35	65

B-10 INJECTION VOLUME: 2µl

B-11 RUN TIME: 20 minutes

B-12 WAVE LENGTH: 230 mm

B-13 PEAK TIME: G Salt — 7.13 minute

B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to G Salt on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of purity G Salt.

$$\% \text{ of G Salt} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \text{X } 100$$

Where

A_1 → Area of Standard G Salt
V_1 → Injection Volume of Standard G Salt
W_1 → Weight of Standard G Salt
B_1 → Total Volume of Standard
A_2 → Area of G Salt peak in Sample
V_2 → Injection Volume of Sample
W_2 → Weight of Sample
B_2 → Total Volume of Sample

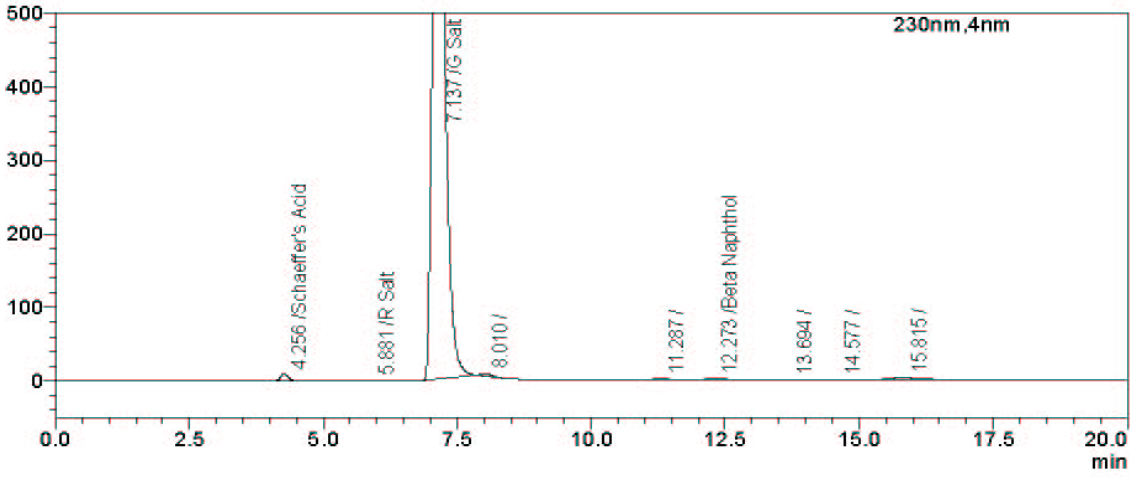


FIG. 1 CHROMATOGRAM

ANNEX C

[*Table* 1 *and Sl No.(iv, v & vi*)]

TO DETERMINE SCHAEFFER'S ACID, R SALT & β –NAPHTHOL IN G SALT BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

C-1 OBJECTIVE

To determine Schaeffer's Acid, R Salt & β-Naphthol in G Salt by high performance liquid Chromatography

C-2 APPARATUS

Binary Gradient Liquid chromatography system with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-3 COLUMN : C18 100A 250 × 4.6mm, 5 µm or equivalent

C-4 REAGENT:

1 Acetonitrile –HPLC grade
2 Water – HPLC Grade
3 Tetra Butyl ammonium Hydrogen sulphate-HPLC Grade
4 G Salt-Known Purity Standard
5 Scheffer's Acid
6 R Salt
7 β –naphthol

C-5 STANDARD PREPARATION:

Weigh accurately 0.050 0 gm standard Schaeffer's Acid in 100 ml volumetric flask dissolve it in Acetonitrile & make up to the mark with Acetonitrile. — Stock Solution A

Weigh accurately 0.050 0 gm standard R Salt in 100 ml volumetric flask dissolve it in Acetonitrile & make up to the mark with Acetonitrile. ─ Stock Solution B

Weigh accurately 0.050 0 gm standard β — naphthol in 100 ml volumetric flask dissolve it in Acetonitrile & make up to the mark with Acetonitrile. — Stock Solution C

Take 10 ml of above stock solution A, B, C in 100 ml volumetric flask & make up to the mark with Acetonitrile — Mixture of Standard solution

C-6 SAMPLE PREPARATION:

Weigh accurately 0.050 0 gm Sample in 100 ml volumetric flask dissolve it in Acetonitrile & make up to the mark with Acetonitrile.

C-7 BUFFER PREPARATION:

0.2% Tetra butyl ammonium hydrogen sulphate in HPLC grade Water.

C-8 FLOW RATE: 1.00 ml/min

C-9 MOBILE PHASE:

Acetionitrile	Buffer
35	65

C-10 INJECTION VOLUME: 2 µl

C-11 RUN TIME: 20 minutes

C-12 WAVE LENGTH: 230 MM

C-13 PEAK TIME: G Salt — 7.13 minute

C-14 CALCULATION

Calculate the peak area of individual constituent pertaining to R Salt on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with standard R Salt.

$$\% \text{ of R Salt} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \text{X } 100$$

Where,

A_1 → Area of Standard R Salt

V_1 → Injection Volume of Standard R Salt

W_1 → Weight of Standard R Salt

B_1 → Total Volume of Standard R Salt

A_2 → Area of R Salt peak in Sample

V_2 → Injection Volume of Sample

W_2 → Weight of Sample

B_2 → Total Volume of Sample

Same calculation for Schaeffer's Acid & β-Naphthol.

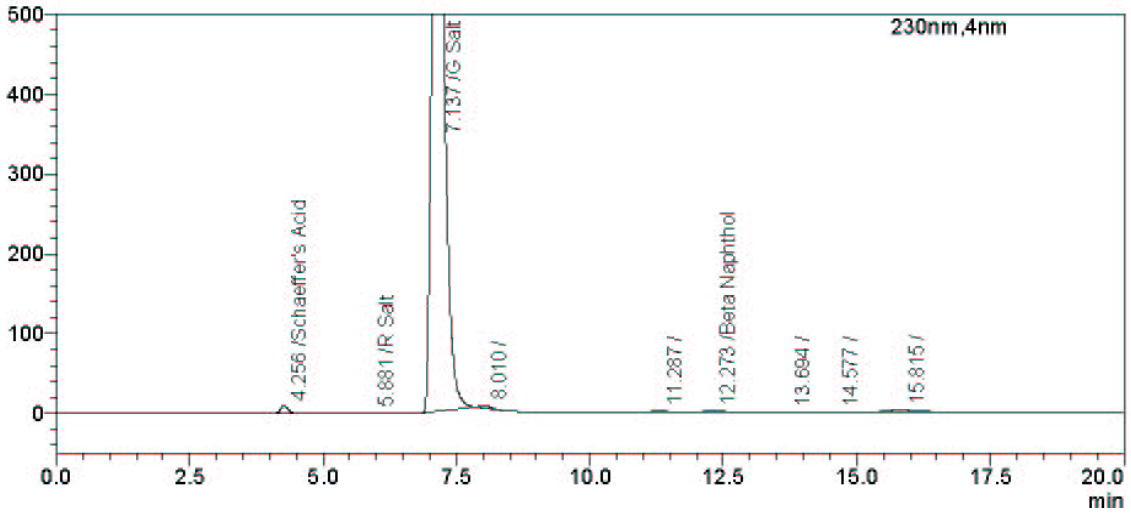


FIG 2 CHROMATOGRAM

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
ICT, Mumbai	Prof P. M. Bhate (***Chairman***)
Aarti Industries Limited, Mumbai	Shri Kirit H. Desai
Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat	Shri Mansukh H. Vekaria
Archroma, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited, Valsad, Gujarat	Dr J. G. Desai Dr M. U. Rahman (*Alternate*)
BASF India Limited, Mumbai	Shri Uday Kulkarni
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K Astik (*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), Ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
Indian Chemical Council, Mumbai	Shri P. S. Singh
Jay Chemical Industries, Ahmedabad	Shri Vilpesh Yadav Shrimati Maitri Vyas (*Alternate*)
Ministry of Chemicals & Fertilizers - Department of Chemicals & Petrochemicals - New Delhi	Shri Jasbir Singh
Ministry Of Defence (Research and Development), New Delhi	Dr Vijay Tak
National Test House (er), Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
Sudarshan Chemical Industries Limited, Pune	Dr Jaychandran M. Nair
The Bombay Textile Research Association	Dr Ashok N. Desai
The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	Shri V. R. Kanetkar
BIS Director General	Shri N. K. Bansal, Scientist 'F' and Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shri Chandrakesh Singh
Scientist 'D', BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 26 (14053).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	2323 7617, 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054	2337 8499, 2337 8561, 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019	265 0206, 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442, 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093	2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi